

The opinion in support of the decision being entered today was not written for publication and is not binding precedent of the Board.

Paper No. 14

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE BOARD OF PATENT APPEALS  
AND INTERFERENCES

Ex parte CHANGMING JIN,  
RICHARD SCOTT LIST,  
and  
JOSEPH D. LUTTMER

Appeal No. 2002-1330  
Application No. 09/178,249

ON BRIEF

**MAILED**

**JUL 16 2003**

**PAT. & T.M. OFFICE  
BOARD OF PATENT APPEALS  
AND INTERFERENCES**

Before GARRIS, TIMM, and DELMENDO, Administrative Patent Judges.  
DELMENDO, Administrative Patent Judge.

DECISION ON APPEAL

This is a decision on an appeal under 35 U.S.C. § 134 (2002) from the examiner's final rejection of claims 1, 2, 5, and 6 (final Office action mailed Jul. 2, 2001, paper 10) in the above-identified application. Claims 3, 4, and 7, which are the only other pending claims, have been "objected to as being dependent upon a rejected base claim" but indicated as allowable

"if rewritten in independent form including all of the limitations of the base claim and any intervening claims."

(Final Office action, page 4.)

The subject matter on appeal relates to a dielectric layer fabrication method. Further details of this appealed subject matter are recited in representative claims 1 and 5 reproduced below:

1. A dielectric layer fabrication method, comprising the steps of:
  - (a) applying a layer of dielectric precursor on a body;
  - (b) flowing a precursor reaction catalyst over said layer; and
  - (c) completing a precursor reaction to form a dielectric layer.
5. The method of claim 1, wherein:
  - (a) said body of step (a) of claim 1 is within a circular cylindrical chamber; and
  - (b) said flowing of step (b) of claim 1 is into said chamber at the circular periphery of said chamber, is radial over said precursor layer to a central axis, and is out of said chamber at said central axis.

The examiner relies on the following prior art references as evidence of unpatentability:

Radhakrishnan	5,650,361	Jul. 22, 1997
Smith et al. (Smith)	5,955,140	Sep. 21, 1999 (filed Nov. 14, 1996)

The appealed claims stand rejected as follows:

- I. claims 1, 2, and 6 under 35 U.S.C. § 102(e) as anticipated by Smith (examiner's answer mailed Mar. 26, 2002, paper 13, page 3); and
- II. claim 5 as under 35 U.S.C. § 103(a) unpatentable over Smith in view of Radhakrishnan (id.).<sup>1</sup>

We affirm both rejections.<sup>2</sup>

Smith describes a method for forming a nanoporous dielectric layer on a semiconductor substrate comprising:

- (a) applying a nanoporous aerogel precursor sol on the substrate, the precursor sol comprising specified amounts of a metal-based aerogel precursor reactant and a specified amount of a first solvent comprising glycerol; and
- (b) gelling (i.e., reacting) the precursor sol and drying to form a dry nanoporous dielectric layer.

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<sup>1</sup> The appellants' statement of the issues (appeal brief filed Dec. 3, 2001, paper 12, p. 2) is erroneous.

<sup>2</sup> The appellants submit: "The claims are treated as single group [sic] in each rejection." (Appeal brief, p. 2.) However, the appellants present only one set of arguments against both prior art rejections and only with respect to claim 1. (Id. at p. 3.) Although the examiner rejected claim 5 separately over the combined teachings of Smith and Radhakrishnan, the appellants deliberately chose to rely on the same arguments for both grounds of rejection. Under these circumstances, we

(Column 6, lines 10-60.) Smith further describes the introduction of a vapor-phase catalyst such as ammonia to further activate the sol and promote rapid cross-linking. (Column 8, lines 46-50; column 30, line 62 to column 31, line 8.)

The appellants' only discernible argument is that "Smith...does not suggest any flowing of the catalyst over the precursor layer as required by claim 1." (Appeal brief, page 3.) We disagree.

To start, we note that Smith's catalyst is described as performing the same function (i.e., catalyze the gelation reaction) as the appellants' recited catalyst. (Specification, page 6, lines 21-23; Smith's column 8, lines 40-53.) Also, while a preferred embodiment of the invention includes "continuously flowing" the catalyst through the "gel aging chambers," this feature of the invention is not specified in appealed claim 1.

As pointed out by the examiner (answer, page 4), the introduction of Smith's catalyst vapor into closed chamber 32 (Figure 19C) to catalyze the reaction (i.e., gelation) of the precursor sol film would necessarily result in the movement of

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confine our discussion to claim 1. 37 CFR §§ 1.192(a), c(7) and c(8) (1995, 1997).

catalyst vapor (e.g., by diffusion) molecules over the precursor sol film, thus satisfying the here claimed limitation of "flowing a precursor reaction catalyst over said layer."<sup>3</sup> In this regard, the fundamentals of mass-transfer dictate that "[w]hen a homogeneous material - either gas, liquid, or solid - contains two or more components whose concentrations vary from point to point, there is a tendency for transfer of mass to take place in such a way as to cause the concentrations to become uniform." See Chemical Engineers' Handbook 14-3, 14-4 (Robert H. Perry & Cecil H. Chilton eds., 5<sup>th</sup> ed. 1973), copy attached. Absent any special definition in the appellants' specification for the term "flowing,"<sup>4</sup> we must uphold the examiner's determination that Smith discloses, either expressly or inherently, each and every limitation of appealed claim 1.

The appellants' arguments regarding Radhakrishnan as they relate to appealed claim 1 are irrelevant, because this claim has been rejected under 35 U.S.C. § 102(e) as anticipated by

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<sup>3</sup> For completeness, we attach a copy of Webster's Third New International Dictionary 875, for the definition of the root term "flow." The examiner's definition of the term (answer, p. 4) is consistent with the meaning of the term as set forth in the dictionary.

<sup>4</sup> In re Morris, 127 F.3d 1048, 1054, 1056, 44 USPQ2d 1023, 1027, 1029 (Fed. Cir. 1997); In re Zletz, 893 F.2d 319, 321-22, 13 USPQ2d 1320, 1322 (Fed. Cir. 1989).

Smith. In any event, the appellants do not dispute the examiner's apparent determination that when the teachings of Smith and Radhakrishnan are combined, one of ordinary skill in the art would have been led to arrive at a method encompassed by appealed claim 1.<sup>5</sup> Rather, the appellants argue that Radhakrishnan does not suggest any "flowing" of a catalyst over a precursor layer. (Appeal brief, page 3.)

We are not persuaded by the appellants' argument. When properly construed, the claim limitation "flowing a precursor reaction catalyst over said layer" includes the type of catalyst vapor movement as described in Smith. The examiner's rejection is based not on Radhakrishnan alone but instead on the collective teachings of both applied prior art references. In re Keller, 642 F.2d 413, 425, 208 USPQ 871, 881 (CCPA 1981) ("The test for obviousness is not whether the features of a secondary reference may be bodily incorporated into the structure of the primary reference...Rather, the test is what the combined

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<sup>5</sup> In the event of continued examination, the examiner should provide a detailed explanation on how the combined teachings of Smith and Radhakrishnan would have suggested to one of ordinary skill in the art a method in which "(b) said flowing of step (b) of claim 1 is into said chamber at the circular periphery of said chamber, is radial over said precursor layer to a central axis, and is out of said chamber at said central axis," as recited in appealed claim 5.

teachings of the references would have suggested to those of ordinary skill in the art." ).

The appellants further argue that Radhakrishnan describes cleaning a substrate prior to deposition of the dielectric layer and does not relate to dielectric layer formation. (Appeal brief, page 3.) According to the appellants (id.), Radhakrishnan does not teach the use of a catalyst and "has no suggestion of either a precursor layer or a catalyst." Again, the appellants incorrectly focus on the teachings of Radhakrishnan alone rather than the collective teachings of both prior art references. For this reason, the appellants' argument fails.

In summary, we affirm the examiner's rejections under: (i) 35 U.S.C. § 102(e) of appealed claims 1, 2, and 6 as anticipated by Smith; and (ii) 35 U.S.C. § 103(a) of appealed claim 5 as unpatentable over Smith in view of Radhakrishnan.


The decision of the examiner is affirmed.

Appeal No. 2002-1330  
Application No. 09/178,249

No time period for taking any subsequent action in connection with this appeal may be extended under 37 CFR § 1.136(a).

AFFIRMED

Bradley R. Garriss  
Administrative Patent Judge

  
Catherine Timm  
Administrative Patent Judge

BOARD OF PATENT

APPEALS AND

## INTERFERENCES

  
Romulo H. Delmendo  
Administrative Patent Judge

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Appeal No. 2002-1330  
Application No. 09/178,249

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REPRODUCED AND HANDED: Handbook of Heat Transfer  
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The editors for this book were Harold B. Crawford and Ross J. Kepler, the designer was Naomi Auerbach, and its production was supervised by Stephen J. Boldish. It was set in Fototronic Laurel by York Graphic Services, Inc.

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Techniques will be presented for determining the necessary time of contact between the flowing streams or the required height of tower.

### EQUILIBRIUM DATA\*

Equilibrium data necessary for absorption calculations include the solubility of the solute gas in the solvent. In order to completely define the solubility, the data must generally state the temperature, the concentration of the solute gas in the liquid phase, the pressure of the solute gas in the gas phase, and the total pressure on the system. At low pressures the total pressure of the system is not so important, but as the total pressure increases, it can have a more significant effect on gas solubility. Equilibrium data will generally be found in one of three forms: solubility data expressed as either solubility in weight or mole per cent or as the Henry's law constant, pure component vapor pressures, or equilibrium distribution coefficients.

**Solubility of Gases in Liquids.** Where Henry's law holds, solubility is defined by giving the Henry's law constant and the temperature  $H = p_a/x_a = \text{atm./mole fraction of solute in solution}$ . For many gases Henry's law holds quite well when the partial pressure of the solute gas is less than 1 atm. For partial pressures of solute gas greater than 1 atm.,  $H$  is seldom independent of the partial pressure of the solute gas. In these instances  $H$  varies with partial pressure, and a given value of  $H$  can be used over only a narrow range of pressures. The use of Henry's law constants in obtaining liquid concentrations from gas solubility data is illustrated in the example below.

**Example 1.** We need to determine how much hydrogen from a gas mixture can be dissolved in 100 lb. of water when the total pressure on the gas is 760 mm. Hg. The partial pressure of hydrogen in the gas mixture is 200 mm. Hg, and the temperature is 20°C.

For partial pressures of hydrogen up to one atmosphere the value of  $H$  is  $6.83 \times 10^4$  at 20°C. ("International Critical Tables," vol. 3, p. 256):

$$x_A = \frac{P_A}{H_A} \quad (14-1)$$

$$P_A = 200 \text{ mm. Hg.} = 0.263 \text{ atm.}$$

$$x_A = \frac{0.263}{6.83 \times 10^4} = 0.385 \times 10^{-5}$$

where  $x_A$  is the mole fraction of hydrogen in the liquid phase. To calculate the pounds of hydrogen per hundred pounds of water we must convert from a molar to a weight basis. In a two-component system, the following formulation may be used:

$$\left( \frac{x_A}{1 - x_A} \right) \left( \frac{m_A}{m_B} \right) 100 = \left( \frac{0.385 \times 10^{-5}}{1 - 0.385 \times 10^{-5}} \right) \left( \frac{2.02}{18.02} \right) 100 = 0.431 \times 10^{-4}$$

Thus,  $0.431 \times 10^{-4}$  lb. (g.) of hydrogen is the maximum that can be dissolved in 100 lb. (g.) of water at 20°C. from a gas mixture under a hydrogen partial pressure of 200 mm. Hg.

Obtaining solubility data for the system under consideration can sometimes be a challenging problem. An idea of the range of solutes and solvents that the chemical engineer may encounter in his absorption problems can be gained by studying Table 14-2. The "International Critical Tables" are always an excellent starting point. Markham and Cobey [*Chem. Rev.*, **28**, 519 (1941)] summarized and critically reviewed gas solubility data available before 1941. Battino and Clever [*Chem. Rev.*, **66**, 395-463 (1966)] review more recent data with emphasis on solvents other than water. Osburn and Markovic [*Chem. Eng.*, pp. 105-108, Aug. 25, 1969] present monographs for determining  $H$  at 20°C. when surface tension and molar volume of the solvent are known.

Solubility data for hydrocarbons and oils are usually presented as pure component vapor pressures or as equilibrium constants ( $K = y/x$ , where  $y$  = mole fraction of the solute in the gas phase, and  $x$  = mole fraction of the solute in the liquid phase). Through the use of Raoult's law ( $pp_A = P_A x_A$ , where  $pp_A$  = partial pres-

Table 14-2. Solute and Non-aqueous Solvents for Gas Absorption

Solutes	Solvents
Acetylene, $C_2H_2$	Acetic acid (glacial), $C_2H_4O_2$
Air	Acetic anhydride, $C_4H_6O_3$
Ammonia, $NH_3$	Acetone, $C_3H_6O$
Bromine, $Br_2$	Amyl alcohol, $C_5H_{12}O$
Carbon dioxide, $CO_2$	Aniline, $C_6H_7N$
Carbon monoxide, CO	Benzene, $C_6H_6$
Chlorine, $Cl_2$	Bromobenzene, $C_6H_5Br$
Ethane, $C_2H_6$	Carbon disulfide, $CS_2$
Ethylene, $C_2H_4$	Carbon tetrachloride, $CCl_4$
Hydrogen, $H_2$	Chlorobenzene, $C_6H_5Cl$
Hydrogen chloride, HCl	Chloroform, $CHCl_3$
Hydrogen sulfide, $H_2S$	Ethyl acetate, $C_4H_8O_2$
Methane, $CH_4$	Ethyl alcohol, $C_2H_6O$
Methyl chloride, $CH_3Cl$	Ethylene chloride, $C_2H_4Cl_2$
Nitric oxide, NO	Ethyl ether, $C_4H_{10}O$
Nitrogen, $N_2$	Methyl acetate, $C_3H_6O_2$
Nitrous oxide, $N_2O$	Methyl alcohol, $CH_3O$
Oxygen, $O_2$	Nitrobenzene, $C_6H_5NO_2$
Sulfur dioxide, $SO_2$	Propyl alcohol, $C_3H_8O$
Etc.	Propylene, $C_3H_6$
	Toluene, $C_7H_8$
	Etc.

sure in gas phase, and  $P_A$  = pure component vapor pressure) vapor pressures can be used to predict solubilities. Extreme care must be taken, however, in attempting to use pure component vapor pressures to predict gas absorption behavior. Both liquid-phase and vapor-phase non-idealities can cause significant deviations from the behavior predicted from pure component vapor pressures combined with Raoult's law. Equilibrium distribution coefficients ( $K$ ) vary with temperature, pressure, and composition, as discussed in Sec. 13. Assuming that data are available for a given system under similar conditions of temperature and pressure, the  $K$  value is a much more reliable tool for predicting vapor-liquid distribution.

Vapor-pressure data are available in Sec. 3 of this handbook for a number of materials. Dreisbach ("Pressure-Volume-Temperature Relationships of Organic Compounds," Handbook Publishers, Sandusky, Ohio, 1952) presents an exhaustive compendium of vapor-pressure data for various families of hydrocarbons.

Values of the equilibrium distribution coefficient  $K$  are given by Katz and Hachmuth [*Ind. Eng. Chem.*, **29**, 1072 (1937)]. See also Sherwood and Pigford ("Absorption and Extraction," McGraw-Hill, New York, 1952) and an extensive series of articles by Sage et al. [*Ind. Eng. Chem.*, 1934 to date]. In addition, the "Engineering Data Book" of the Natural Gas Processors Association in both the 1957 and 1966 editions presents significant data, as do Winn [*Petrol. Refiner*, **33**(4), 132 (1954)], Hadden and Grayson [*Hydrocarbon Process. Petrol. Refiner*, **40**(9), 207 (1961)], and Grayson and Streed (6th World Petroleum Congress, 1963). Chao and Seader [*A.I.Ch.E. J.*, **7**, 598-605 (1961)] have presented a wide-ranging correlation which takes into account both the vapor-phase and liquid-phase non-ideality effects up to pressures in the neighborhood of 1500 p.s.i.a.

In the case of absorption combined with chemical reaction, the engineer must be particularly careful that the data he uses include the effect of incomplete stripping on the absorption process. Burns and Maddox [*Oil Gas J.*, **65**, 112 (Sept. 18, 1967)] used data from Kohl and Reisenfeld ("Gas Purification," McGraw-Hill, New York, 1960) to solve in detail a problem illustrating the effect of mutual solubilities and incomplete stripping on absorption of  $H_2S$  and  $CO_2$  in an amine-water solution.

### MASS-TRANSFER FUNDAMENTALS

**Homogeneous Diffusion.** When a homogeneous material—either gas, liquid, or solid—contains two or more components whose concentrations vary from point to point, there is a tendency for

\*Equilibrium solubility data for specific systems are given in Sec. 3.

transfer of mass to take place in such a way as to cause the concentrations to become uniform. This phenomenon is associated with the thermal agitation of molecules; in a region where molecules of one kind are concentrated, there is a greater tendency for molecules of this kind to escape than to enter the region. The net rate of diffusion  $N_A$  of material A at a point in a stationary fluid is found from experiment as well as from theory to be proportional to the concentration gradient at the point,

$$N_A = -D_p \frac{\partial c}{\partial s} \quad (14-2)$$

where  $c$  = concentration,  $s$  = distance, and  $D_p$  = diffusivity. If  $c$  is expressed in g.-moles/cu. cm.,  $s$  in cm., and  $D_p$  in sq. cm./sec., then the units of  $N_A$  are g.-moles/(sec.)(sq. cm.). The rate of diffusion is rapid in gases and much slower in liquids.

In the application of the theory of diffusion, it is often desirable to employ integrated forms of the diffusion equation, rather than Eq. (14-2), which is applicable only at a single point. Treatments of the use of Eq. (14-2) for steady-state diffusion are given by Treybal ("Mass-transfer Operations," McGraw-Hill, New York, 1955) and by Sherwood and Pigford ("Absorption and Extraction," McGraw-Hill, New York, 1952).

Several integrated forms of Eq. (14-2) are presented below, along with integrated expressions for the analogous equation for unsteady-state diffusion [Eq. (14-6)]. All these relationships are based on the assumption that diffusivity is not dependent on concentration. This assumption is good for gaseous systems (except at high pressure) and for dilute liquid solutions, but may not be true for concentrated solutions. Dependence on concentration is the result of (1) change of mobility of the solute with concentration because of a change in average molecule size of the medium; and (2) deviations of the mixture from ideal behavior [Wilke, *Chem. Eng. Progr.*, 45, 218 (1949)].

**Steady-state Equimolar Counterdiffusion.** This case is typified by the mixing of two gases in a confined space and by counterdiffusion of two components in distillation. For this case, assuming  $D_p$  constant, Eq. (14-2) integrates to

$$N_A = \frac{D_p}{B_p} (c - c_i) = \frac{D_p}{RTB_p} (p - p_i) = \frac{D_p P}{RTB_p} (y - y_i) = k_G (y - y_i) \quad (14-3)$$

where concentration  $c$  can be expressed alternatively in terms of partial pressure  $p$  or mole fraction  $y$ . In c.g.s. units,  $N_A$  = g.-moles/(sec.)(sq. cm.);  $D_p$  = sq. cm./sec.;  $c$  = g.-moles/cc.;  $p$  = atm.;  $B_p$  = layer thickness, cm.;  $R$  = universal gas constant, 82.06(cc.)(atm.)/(g.-mole)(°K);  $T$  = temperature, °K;  $P$  = absolute pressure, atm.;  $k_G$  = gas-phase mass-transfer coefficient, g.-moles/(sec.)(sq. cm.)(mole fraction). Subscript  $i$  refers to the interface. Other consistent sets of units may be used with suitable adjustments in the numerical value of  $R$ .

**Steady-state Diffusion of One Component through a Second Stagnant Component.** Examples are absorption of a soluble gas from a second insoluble gas and absorption of a slightly soluble gas into a non-volatile liquid. The integrated equation is

$$N_A = \frac{D_p P}{RTB_p (1 - y_{lm})} (y - y_i) = k_G (y - y_i) \quad (14-4)$$

where  $k_G$  = gas-phase mass-transfer coefficient corrected for inert gas concentration =  $k_G(p_{BM}/P)$ ;  $p_{BM}$  = partial pressure of inert gas;  $lm$  = logarithmic mean; other symbols as defined above.

**Steady-state Diffusion of One Component through a Stagnant Multicomponent Mixture.** According to Wilke [*Chem. Eng. Progr.*, 46, 95 (1950)], Eq. (14-1) may be applied to this case provided an effective diffusivity of the diffusing species A is defined as

$$D_{eA} = \frac{1 - y_A}{(y_B/D_{AB}) + (y_C/D_{AC}) + (y_D/D_{AD}) + \dots} \quad (14-5)$$

**Unsteady-state Diffusion.** Diffusion does not lead to conditions of constant concentration gradient unless a steady state is estab-

lished. It is therefore often necessary to consider the change of concentration  $c$  with time  $t$  caused by diffusion as represented by the differential equation

$$\frac{\partial c}{\partial t} = D_p \frac{\partial^2 c}{\partial s^2} \quad (14-6)$$

where  $s$  = distance and  $D_p$  = diffusivity.

Solutions of this equation for a diversity of physical situations are given by Crank ("Mathematics of Diffusion," Oxford, New York, 1956) and by Jost ("Diffusion," Academic Press, New York, 1952). Figure 14-1 shows the change in average concentration  $\bar{c}$  of a component in a slab, cylinder, or sphere as a function of time  $t$  when a constant surface concentration  $c_i$  is provided to permit that component to diffuse (i.e., where the relative resistance to diffusion in the surrounding medium is negligible). The solution is analogous to that for the conduction of heat under the influence of a temperature gradient. Figure 14-1 is applicable only when the material in which the diffusing component is dispersed is not internally mixed and retains its shape during the period of time involved. This is not the case in packed or plate-column gas absorbers but may be so in some spray, descending-liquid-sheet, or falling-jet devices if the fluid is stagnant or in laminar flow.

**Diffusion with Flow.** If fluid motion is laminar, transfer of mass between adjacent layers of fluid takes place purely by molecular diffusion. If the velocity pattern of the flow is known, it is sometimes possible to calculate the over-all rate of mass transfer into the moving fluid by the use of the basic equations of molecular diffusion. If the flow is turbulent, however, such calculations are generally impossible, since the laws that govern the transport of matter by turbulent mixing of small volumes of fluid are not well enough understood. Prediction of mass-transfer rates under such conditions is based on empirical methods.

**Laminar Flow, Uniform Velocity.** If the velocity of a flowing stream is uniform over a very deep region (thickness  $B_p \gg \sqrt{D_p t}$ ) in which diffusion is taking place, Eq. (14-6) is applicable. It has

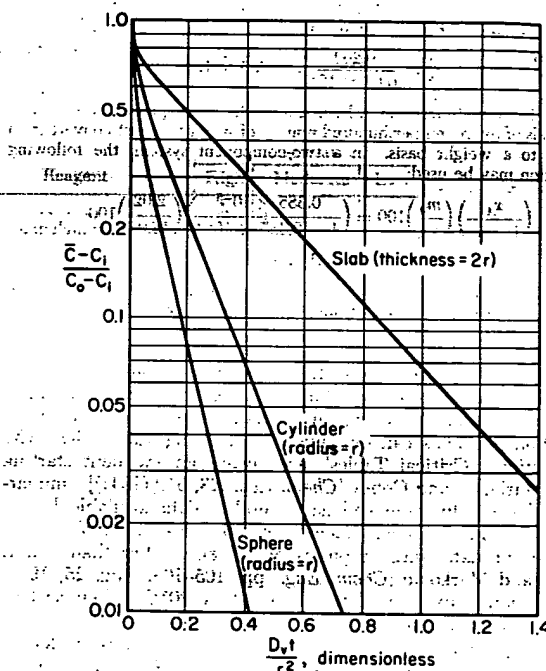


FIG. 14-1. Unsteady-state diffusion:  $c_o$  = uniform initial concentration,  $c_i$  = constant surface concentration,  $\bar{c}$  = average concentration. (Crank, "Mathematics of Diffusion," Oxford, Clarendon, 1956.)

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